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Experimental investigation on the carbide precipitation and mechanical property evolution of a cryogenically treated tool steel

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Abstract

In this present paper, the evolution of the carbide precipitation, hardness and impact energy of semi knocked down (SKD) 11 tool steel submitted to three different cryogenic treatment processes were investigated with respect to those that experienced conventional heat treatment. The cryogenic treatment processes in this study were conventional cold treatment, shallow cryogenic treatment, and deep cryogenic treatment. The evolution of carbide precipitation was examined using optical microscope and X-ray diffraction. Hardness evolution of all treated specimens was investigated using the Rockwell hardness test. Charpy V-notch impact test was employed to evaluate the impact toughness energy of all treated specimens. The results show that cryogenic treatment resulted in an increased amount of secondary carbides and the precipitated secondary carbides were observed to be considerably higher in deep cryogenically treated SKD 11. Hardness was enhanced in specimens subjected to cryogenic treatments, especially in deep cryogenic treatment. The decreased impact toughness of cryogenically treated specimens was found and this could be used to reflect the decreased amount of retained austenite and the increased amount of secondary carbides.

Keywords: Cryogenic treatment, SKD 11 tool steel, Carbide precipitation, Mechanical properties

1. Introduction

As technology rapidly progressed, the highly-efficient tool steels are required to machine a number of the hard-to-machine materials at the higher rate of cutting with a reliable metal-forming performance [1,2]. To deal with such demand, the performance of tool steels, especially in terms of hardness, needs to be improved [2,3]. Normally, tool steels can be hardened by conventional heat treatment. Conventional heat treatment is known as a common practice which can be performed by heating and holding a certain tool steels in a controlled environment for a specific period of time at a certain temperature before cooling down to induce the desired properties of tool steels [3,4]. Nevertheless, this thermal treatment has its own limitation for tool steels because it can potentially produce a soft matrix and coarse carbides. Thus, this treatment cannot be used to fully enhance the metal-forming performance of tool steels [5,6].

Since the last century, the add-on process of this common heat treatment, known as cryogenic treatment, has been studied. Basically, cryogenic treatment can be carried out by cooling down the hardening steels to a sub-zero temperature and then soaking them at this temperature for a period of time [6,7]. Technically, this method can be divided into three different temperature processes [5-8]. The first one is conventional cold treatment whereby the temperature is decreased to -80 ˚C. The second one is shallow cryogenic treatment, which is conducted in the temperature range of -80 to -160 ˚C. The last one is deep cryogenic treatment, which is performed below -160 ˚C. In practice, cryogenic treatment is performed to convert the retained austenite into martensite, resulting in the removal of residual stress, enhanced dimensional stability and improved wear resistance of the steel [2-8]. The mechanisms of the retained austenite transformation are widely discussed with regard to this enhanced wear resistance of tool steels. However, information about carbide precipitation is still lacking. Besides, for the further improvement of this process, a better understanding of the properties, such as the hardness and toughness, should be presented in more detail.

In this paper, the results from the carbide precipitation investigation using optical microscopy and X-ray diffraction (XRD) are discussed. The evolution of the hardness and the variation of the impact energy are presented and discussed. One of the aims of the investigation is to investigate the effect of cryogenic treatment on the microstructural evolution of a particular tool steel; another is to obtain information on the evolution of hardness and impact toughness of a cryogenic treated tool steel. The tool steel selected in this study is SKD 11, which is widely used as commercial punches and dies [1]. Thus, the obtained results should provide useful information for the development of tool steels in metal-forming industries.

2. Materials and methods

SKD 11 tool steel, with the chemical compositions as shown in Table 1, was used in this investigation because it represents one of the most common tool steels used for typical metal-forming processes [1].

Table 1 Chemical compositions of the A356 alloy used in this study (in %wt).

Sample	C	Si	Mn	Cr	Mo	V	P	S
SKD11	1.4-1.6	< 0.4	< 0.6	11-13	0.8-1.2	0.2-0.5	< 0.0.3	< 0.03
Specimens	1.52	0.38	0.47	12.1	1.1	0.42	0.027	0.28

From Table 1, the chemical compositions of the specimens obviously comply with the Japanese Industrial Standard (JIS) specification of SKD 11 steel. Specimens were prepared with a size of 40 mm × 85 mm × 16 mm. They were submitted to conventional heat treatment and three cryogenic treatment conditions in independent sets. The conventional heat treatment involved quenching and tempering, whereas cryogenic treatment was related to a supplementary sub-zero treatment sequence which was intermediately conducted after quenching. In this experiment, SKD 11 tool steel specimens submitted to conventional cold treatment, shallow cryogenic treatment and deep cryogenic treatment were systematically investigated and compared with those that were submitted to conventional heat treatment. All of the selected cryogenic treatments involved sub-zero treatment conditions set up at -50, -100 and -190 ˚C, respectively. All of the treatment conditions are schematically given in Figure 1.

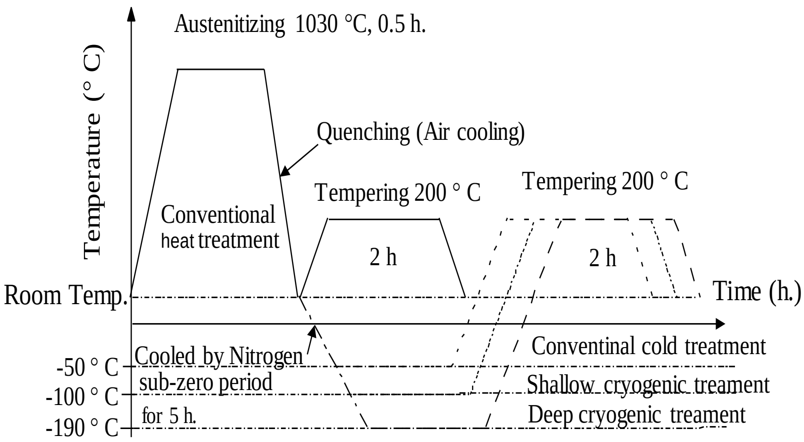


Figure 1 Treatment processes of SKD 11 specimens in this experiment.

For conventional heat treatment, specimens were austenitized at 1,030 ˚C for 30 min and then cooled by air cooling. For cryogenic treatments, air-cooled specimens were sub-cooled in the self-made liquid nitrogen treatment system, the details of which are given in Figure 2. The cryogenic temperature conditions in this experiment were set at -50, -100 and -190 ˚C for 5 h, which are in accordance with the sub-zero conditions for high-carbon tool steels suggested from the works of Bensely et al [9,10]. All of the treated specimens were tempered at 200 ˚C for 2 h to relieve the residual stress due to the heat treatment process [11]. Temperature conditions of all treatment processes were kept constant with a precision of ± 1 ˚C. The rate of heating and cooling were controlled at 1 ˚C/min.

For metallographic examination, the treated specimens were ground with silicon carbide (SiC) paper down to 1,200 grade emery paper and then polished using a diamond suspension. The etchant was comprised of a mixture of 100 mL distilled water, 10 mL hydrochloric acid (65% conc.), and 1 g potassium metabisulphite. After being cleaned with ethanol, treated specimens were kept in a desiccator for 24 h. Microstructural examination was then carried out using light optical microscopy. XRD with a Cu K-alpha target was operated at 50 kV and 250 mA to obtain XRD patterns of treated specimens at a scanning speed of 2 $^{\circ}$ C/min^{-1}. The estimation of the amount of carbides was then carried out using the Rietveld method with TOPAS software (version 4.2). The hardness test was performed on all treated specimens prepared in accordance with JIS Z2245 standard [12] using MMT-X3A Rockwell hardness tester. This test was carried out with a 150 kg for major load with a dwell time of 15 sec. It was carefully conducted to obtain the average hardness value of treated specimens, estimated from at least 5 measurements. Charpy V-notch impact tests were carried out to evaluate the impact toughness energy at room temperature of all treated specimens. The specimens for the impact toughness test were prepared according to JIS Z2242 standard [13]. Both the hardness and impact toughness energy that were measured can be used to indicate the variation in the amount of retained austenite and precipitated carbides at the different treatment conditions.

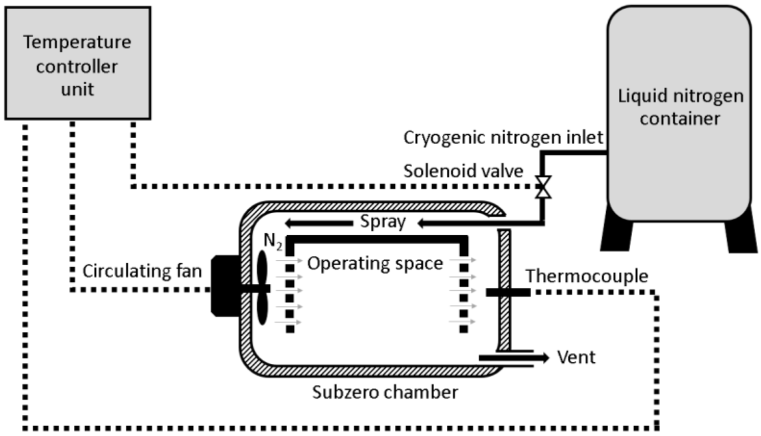


Figure 2 Schematic diagram of the self-made liquid nitrogen treatment system.

3. Results and discussion

3.1 Microstructural Examination

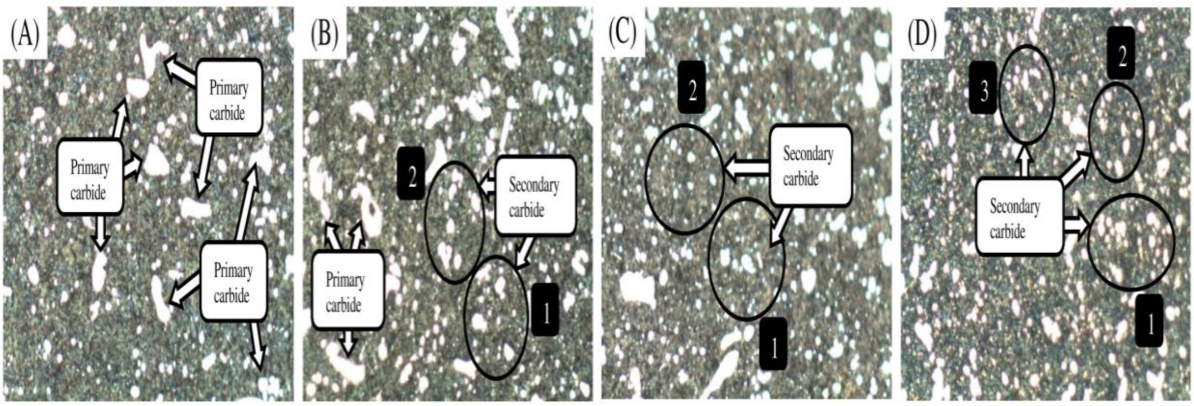


Figure 3 Micrographs of specimens (at 50 × magnification): (A) Conventional heat treatment; (B) Conventional cold treatment; (C) Shallow cryogenic treatment; and (D) Deep cryogenic treatment.

Figure 3 (A) shows an optical micrograph of the conventionally heat-treated specimen. This micrograph obviously exhibits a non-uniform distribution of coarse carbides on the matrix of the specimen. As indicated by the arrows in Figure 3 (A), the large and elongated-shaped white region in Figure 3 (A) represents the coarse primary carbides on the matrix of the specimen. Figure 3 (B) depicts an optical micrograph of a conventional cold treated specimen. This figure shows the appearance of secondary carbides which are finer than the primary carbides. As indicated by circle 1 and 2 in Figure 3 (B), these carbides were seen to be dispersed throughout the matrix. A comparison of Figure 3 (A) and Figure 3 (B) clearly suggests that conventional cold treatment promoted the formation of finer secondary carbides. Figure 3 (C) displays an optical micrograph of the shallow cryogenically treated specimen. The result in this figure reveals that more secondary carbides were precipitated from the matrix, as shown in circle 1 and 2 in Figure 3 (C). Figure 3 (D) illustrates the optical micrograph of the deep cryogenically treated specimen. This micrograph shows the presence of higher-density secondary carbides, as displayed in circle 1, 2 and 3 in Figure 3 (D). Obviously, cryogenic treatment after the conventional hardening immediately facilitated the evolution of secondary carbides and further decreased the sub-zero temperature from conventional cold treatment to shallow cryogenic treatment; deep cryogenic treatment potentially promoted more precipitated secondary carbides, which are fine in nature.

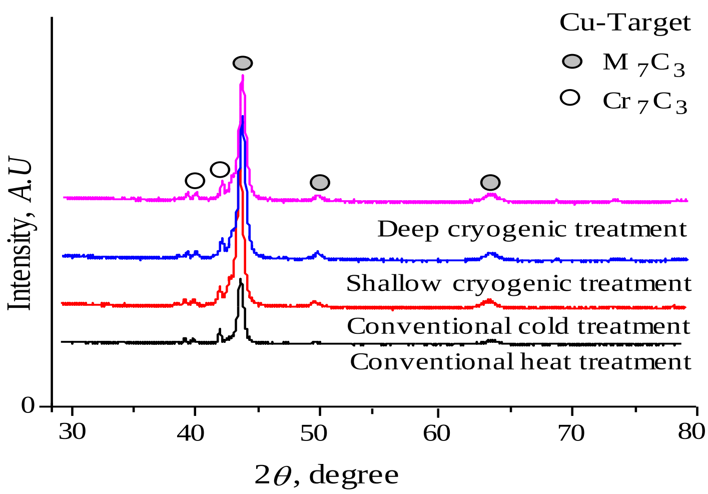


Figure 4 XRD patterns of treated specimens.

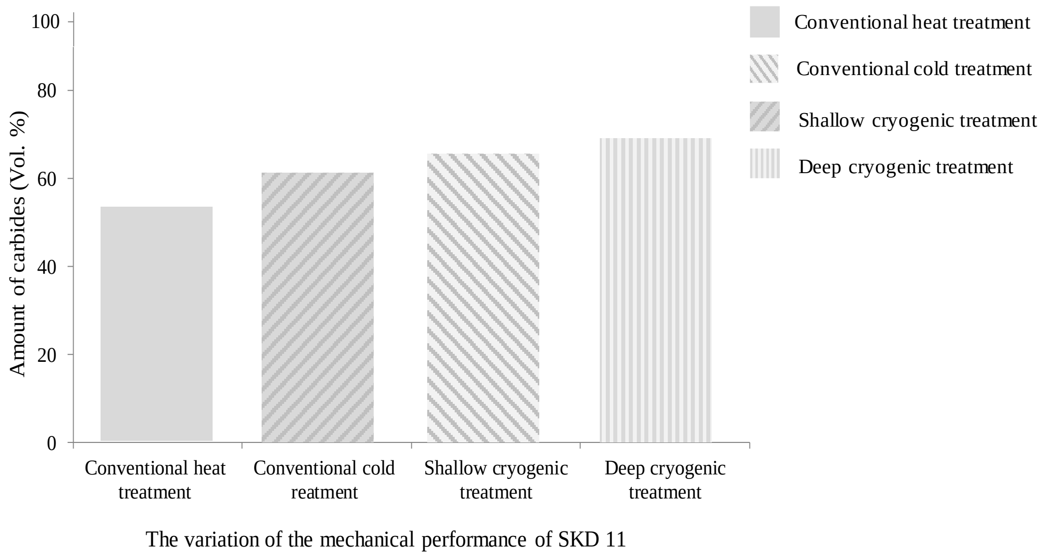


Figure 5 Estimated amount of carbides of different heat-treated SKD 11 specimens.

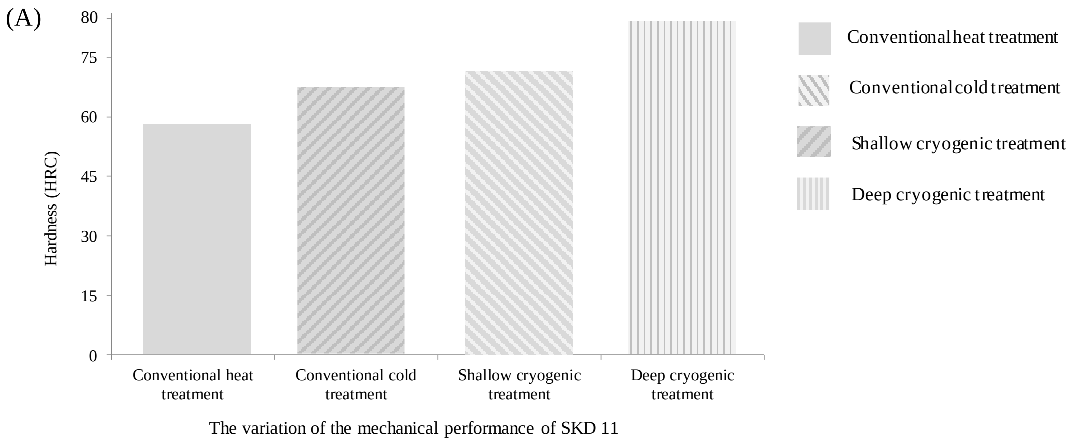
Figure 4 exhibits the XRD profiles of specimens of SKD 11 which were exposed to different treatment conditions. The XRD patterns shown in Figure 4 clearly suggest the presence of M_7C_3, where M could be V, Cr, Mo, and Fe, and Cr_7C_3 in all treated specimens [14-17]. The increased intensity for the diffraction peak of M_7C_3 was clearly found in three cryogenically treated samples, particularly in deep cryogenically treated samples. From a comparison between the optical micrographs in Figure 3(B)-(D) and the XRD patterns in Figure 4, it can be pointed out that an increase in the intensity of the diffraction peak of carbide reflected the precipitation of secondary carbides. In order to conduct further analysis, the amount of carbide was then calculated by using XRD and the help of the Rietveld method with TOPAS software (version 4.2).

Figure 5 shows the effects of the sub-zero treatment on the variation of the estimated amount of carbides. All cryogenic treatment types resulted in an increase in the amount of secondary precipitated carbides when compared to conventional heat treatment. Evidently, the increase in the amount of carbides is found to be more significant in deep cryogenically treated specimens than in shallow cryogenically and conventional cold treated specimens. This finding may also suggest that the secondary carbide density increased in the following order of cryogenic processes: conventional cold treatment, shallow cryogenic treatment, and deep cryogenic treatment. Thus, these results evidently indicate that cryogenic treatment promotes the evolution of secondary carbides and suggest that deep cryogenic treatment particularly accelerated the precipitation of secondary carbides, resulting in the highest carbide density in the microstructures.

3.2 Hardness and impact test

In order to gain a deeper insight in the relationship between the microstructure and mechanical performance of the cryogenic treatment, hardness and impact tests were conducted on the prepared specimens in order to study the evolution of the mechanical performance. Figure 6 shows the effects of the cryogenic treatment on the variation of the mechanical performance of SKD 11 obtained from all treatment types. Figure 6 (A) presents a comparison of the hardness values of SKD 11 specimens that were subjected to different heat treatments. As compared to the conventional heat treatment, all kinds of cryogenic treatments increased the hardness of the specimens, especially in case of the deep cryogenically treated specimen. Technically, the amount of carbides and their density play a major role in the variation of the hardness value of steels [15-17]. Thus, an increase in the hardness value of cryogenically treated specimens in Figure 6 (A) can be contributed to the increased density of fine secondary carbides. The highest hardness values found in the deep cryogenically treated specimen can also be attributed to the highest density of precipitated secondary carbides distributed in the microstructures of specimens obtained from the deep cryogenic treatment. Figure 6 (B) shows the impact energy absorbed in the specimens for all treatment conditions. Basically, impact energy can be considered as an indicator of a material’s toughness. In cases of SKD 11 tool steel, its microstructure normally consists of retained austenite, carbides, and martensite [17]. From a metallurgical perspective, hardness and toughness of steel are directly dominated by the microstructural variation of steel. Generally, the amount of retained austenite is responsible for the soft matrix, but the amount of carbides is related to the hardness of steels [18]. Hence, both of these properties can be reflected by the measurement of impact energy value. Figure 6 (B) illustrates that all cryogenic treatment types tended to decrease the absorbed impact energy of specimens. Cryogenic treatment potentially leads to an increase in precipitation of higher amount of small carbides, as clearly shown in Figure 3- Figure 5. In addition, a decrease in the impact toughness of steels means the reduction of retained austenite [19,20]. Therefore, the decreased impact toughness can be ascribed to the generation of the higher amount of fine secondary carbides and the reduction of the amount of retained austenite, which is influenced by the cryogenic treatment.

The results from this present research indicate that the cryogenic treatment can promote the formation of secondary carbides. These kinds of results have also been found in detailed investigations on experiments of other tool materials. Collins and Dormer [21] studied the evolution of microstructure and hardness of a high chromium tool steel submitted to different cryogenic treatment conditions. They pointed out that the high density of precipitated secondary carbide caused by cryogenic treatments accounted for the increased hardness. Liu et al. [22,23] examined the influences of sub-zero treatment on the microstructural changes and wear of a chromium-containing cast iron. Their results showed that a reduction of residual austenite and increase in fine secondary carbides enhanced the hardness and wear resistance of this cast iron. Zhirafar et al. [24] pointed out that cryogenic treatment enhances hardness but lowers impact resistance of 4340 alloy steel. Rhyime et al [25] and Wierzyllowski et al. [26] indicated that cryogenic treatment, particularly for deep cryogenic treatment, improves hardness properties but decreases the toughness of a cold work tool steel. Evidently, the results from this experiment are agreeable with these commonly accepted works.



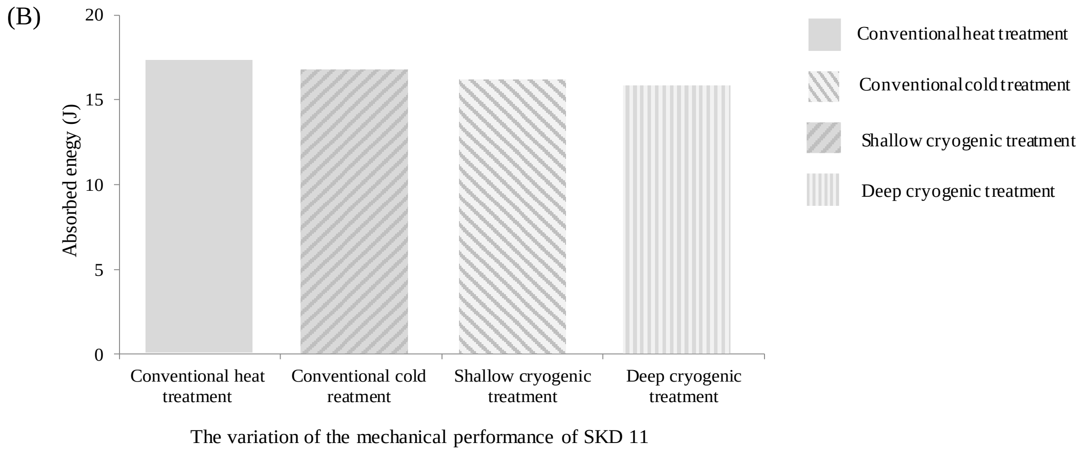


Figure 6 Mechanical performance of different heat-treated SKD 11 specimens: (A) hardness; and (B) impact toughness.

3.3 The hardening evolution mechanism

Cryogenic treatment has been known as a supplementary process which can be added between the hardening and tempering processes of conventional heat treatment. Based on the results of this present work, Figure 7 displays a schematic diagram illustrating the hardening evolution of SKD 11 tool steel submitted to cryogenic treatment.

For SKD 11 steel treated with conventional heat, its microstructure is found to contain a small amount of fine carbides and a large amount of retained austenite. This kind of microstructure cannot fully enhance the hardness of SKD 11 steel. When cryogenic treatment is performed on SKD 11 tool steel, the microstructure is modified. Conventional cold treatment causes the formation of secondary carbides, which are fine in nature, as shown in Figure 3 (B) and Figure 5. In this treatment, SKD 11 tool steel still contains retained austenite with more secondary carbides precipitated from the matrix. Thus, the hardness of SKD 11 tool steel increases. As shallow cryogenic treatment is performed, more secondary carbides are precipitated from the matrix, resulting in an increased density of fine carbides, as indicated in Figure 3 (C) and Figure 5. Besides, the amount of retained austenite decreases. Hence, hardness significantly increases.

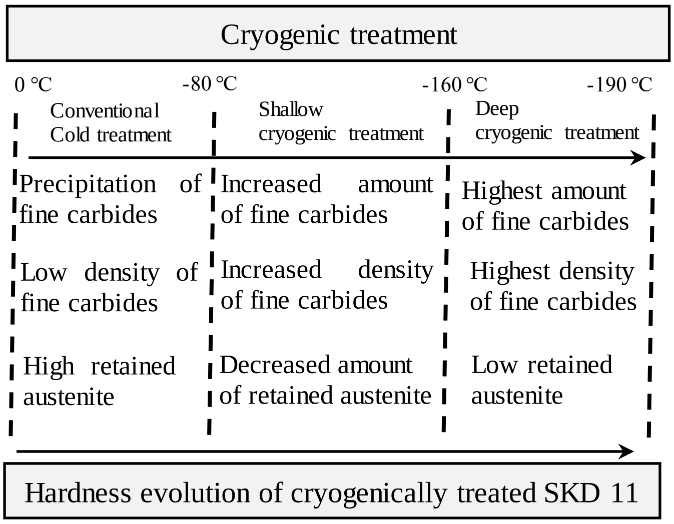


Figure 7 Schematic diagram illustrating the hardening evolution of SKD 11 tool steel submitted to cryogenic treatment.

Deep cryogenic treatment remarkably promotes the formation of secondary carbides, resulting in the highest amount and density of fine carbides, as illustrated in Figure 3 (D) and Figure 5. At the same time, the amount of retained austenite clearly decreases, as implied in Figure 6 (B). Therefore, the highest amount and density of fine carbides and the lowest amount of retained austenite result in the highest degree of hardness of the deep cryogenically treated SKD 11, as seen in Figure 6 (A).

4. Conclusion

The evolution of the carbide precipitation and mechanical property evolution of SKD 11 tool steel submitted to three different cryogenic treatments were systematically investigated in comparison to those subjected to conventional heat treatment. The results from the microstructural evolution investigation showed that cryogenic treatments increased the amount of secondary carbides in the following order of cryogenic processes: cold treatment, shallow cryogenic treatment, and deep cryogenic treatment. The precipitation of secondary carbides was found to be notably higher in specimens subjected to deep cryogenic treatment than those subjected to conventional cold or shallow cryogenic treatment. The results from the mechanical property examination revealed that the hardness of SKD 11 tool steel was improved by the cryogenic treatment, particularly deep cryogenic treatment. The decreased impact toughness of cryogenically treated specimens was observed and this would indicate the decreased amount of retained austenite and the increased amount of secondary carbides.

5. Acknowledgements

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